

High RRR Niobium Material Studies

Ganapati Myneni and Peter Kneisel
Jefferson Lab
Newport News VA23606

Introduction:

Superconducting rf cavities used as accelerating structures in particle accelerators are made from high purity niobium with RRR values of $RRR > 250$. This purity corresponds to a thermal conductivity at 4.2 K of app. 60 W/ m K. The major impurities in this type of niobium are interstitially dissolved gases such as hydrogen, nitrogen, and oxygen in addition to carbon. Hydrogen is readily dissolved in niobium and diffuses at room temperature into the bulk at app. several mm per hour, if the natural oxide layer, which forms a diffusion barrier, is defective. The concentration of hydrogen is somewhat higher at the surface of the material since it has the tendency to cluster at dislocations. At room temperature and concentrations < 6 at % there exists the α phase; at higher concentrations or lower temperatures ordered phases form, especially the ϵ - phase (at $T < 200\text{K}$), which can distort the niobium lattice so much that Niobium is superconducting only at $T < 1.3\text{K}$. In superconducting cavities this "phenomenon" of precipitation of the ϵ - phase is called "Q - disease" and it is reflected in a significant degradation of the Q-value of a superconducting niobium cavity (increased losses by a factor of up to 100 have been observed).

The Q- degradation can be avoided by cooling a cavity quickly through the dangerous temperature region of $75\text{K} < T < 150\text{K}$ thus eliminating the precipitation of the ϵ - phase. However, this is not a permanent "fix"; the only known permanent cure is degassing of the hydrogen from the niobium to low ppm levels. Because of the need for hydrogen degassing the niobium cavities are heat treated at a temperature above 500 C; at 800 C the hydrogen is degassed readily; however it was recently realized with SNS cavities, that a heat treatment temperature of 800 C for a duration of even 1 hour degraded the mechanical properties of the material, in particular decreasing the yield strength. This resulted in the cavities being soft and prone to deforming if they were not handled very carefully, thus affecting the frequency and field profile.

A careful test program was launched to find out the temperature at which the niobium did not change its mechanical properties, but most of the hydrogen present (contributor to the Q-disease) was expelled. Baking at 600 C seems to be appropriate. We baked SNS cavity #4 at 600 C for 10 hrs and saw no evidence of Q-disease¹. In this tech note the usual mechanical properties (yield strength, tensile strength, percentage elongation and Vickers hardness) of the heat treated high RRR niobium at different temperatures are summarized.

Mechanical Properties:

Mechanical properties of high purity niobium have been extensively investigated at JLAB during the period 1988 through 1994, and a summary of this database was presented at the DESY TESLA Workshop, in March 1995, on Cavity Fabrication Techniques at the request of workshop organizers². Most of our work carried out at JLAB was published in various reports and is summarized here for reference. Ambient and cryogenic mechanical properties of the as-received CEBAF production niobium including the reactor grade (Cabot) and high RRR niobium (Teledyne Wah Chang, Heraeus and Fansteel) were presented on invitation at the 1990 State of the Art Electron Beam Melting and Refining Conference³.

Extensive cryogenic mechanical properties of as-received and post-purified Teledyne and Fansteel niobium with RRR over 250 and material thickness of 3.175 mm were reported at the 1993 International Cryogenic Materials Conference⁴. Stress-Strain properties of niobium treated under different conditions such as "as received", post-purified at 1400 C, welded and post-purified at 1400 C, and drastic elongation variation of the post-purified high RRR niobium with BCP and BCP-pressed conditions were incorporated in the first book on RF Superconductivity for Accelerators⁵. Further, thermal and mechanical properties of electron beam welded and heat-treated TESLA niobium were reported in the proceedings of the Sixth workshop on RF Superconductivity organized by JLAB in 1993⁶.

During 1995 it was realized that we have to systematically study the variation of mechanical properties of high RRR niobium with heat treatments at various temperatures and duration of heat treatment since they vary a lot from vendor to vendor and batch to batch². Unfortunately, however, this work was discontinued at that time and the investigations have to be started again because of the problems encountered during the heat treatment of SNS prototype cavities. In addition, to the usual mechanical properties we also report here the Vickers hardness measurements on RRR niobium after various heat treatments ranging from 600 to 800 C for different periods between 1 to 10 hours. Vickers hardness has been included in the Specifications for RRR niobium for SNS since there are indications that this property is related to the degree of recrystallization of the niobium¹⁰.

Results and Discussions:

Yield, tensile strength, percentage of elongation and Vickers hardness

The details of the sample preparation and mechanical properties measurement procedure were described elsewhere⁴. Figures 1, 2 and 3 summarize the effect of heat treatment time and temperature on the mechanical properties of three different batches of SNS prototype niobium WCL (RRR~400), high RRR niobium WC (RRR~400) and RRR niobium TD (RRR~300). The first two batches of RRR~400 niobium were supplied by the same vendor whereas the third batch of niobium of RRR~300 was supplied by a second vendor.

Table I is the summary of the yield strength, tensile strength and percentage of elongation of the various measured samples. The sample numbers ending with ASR#

are as received samples and the numbers such as 6006 means the sample is heat treated at 600 C for 6 hours etc.

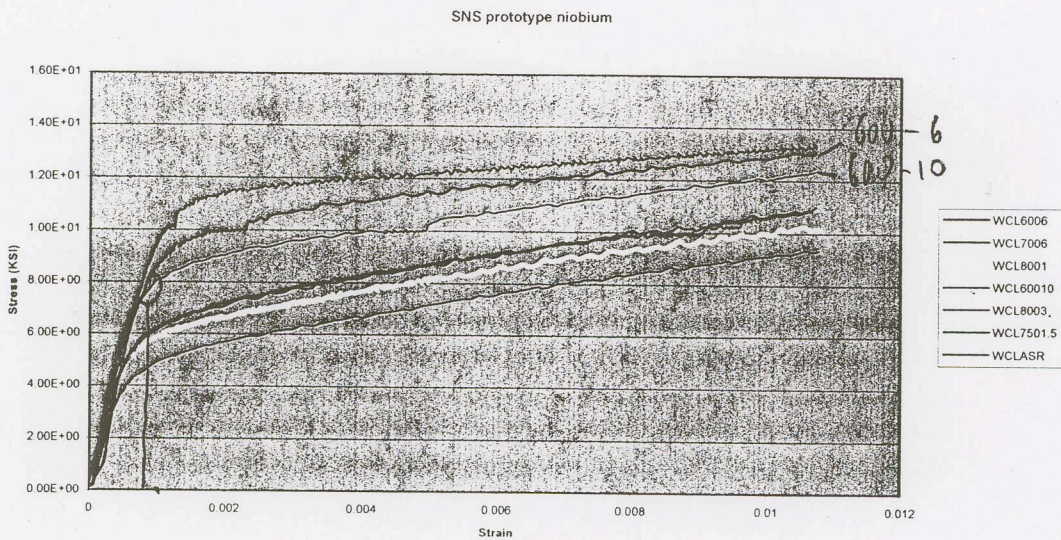


Figure 1(a) Stress - Strain curves with different heat treatments

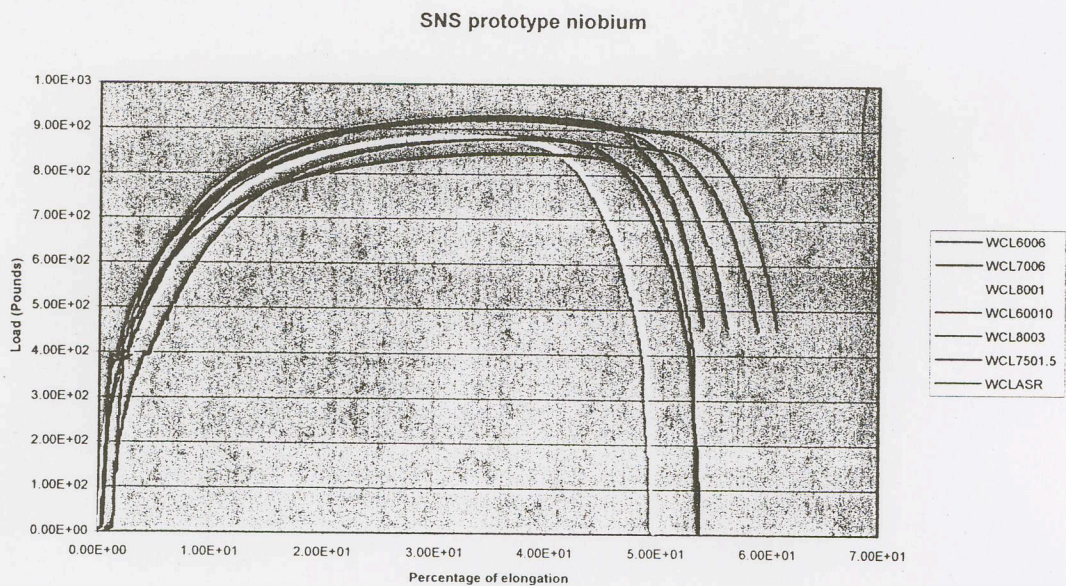


Figure 1(b) Load - Percentage of elongation for different heat treatments

SNS high RRR niobium

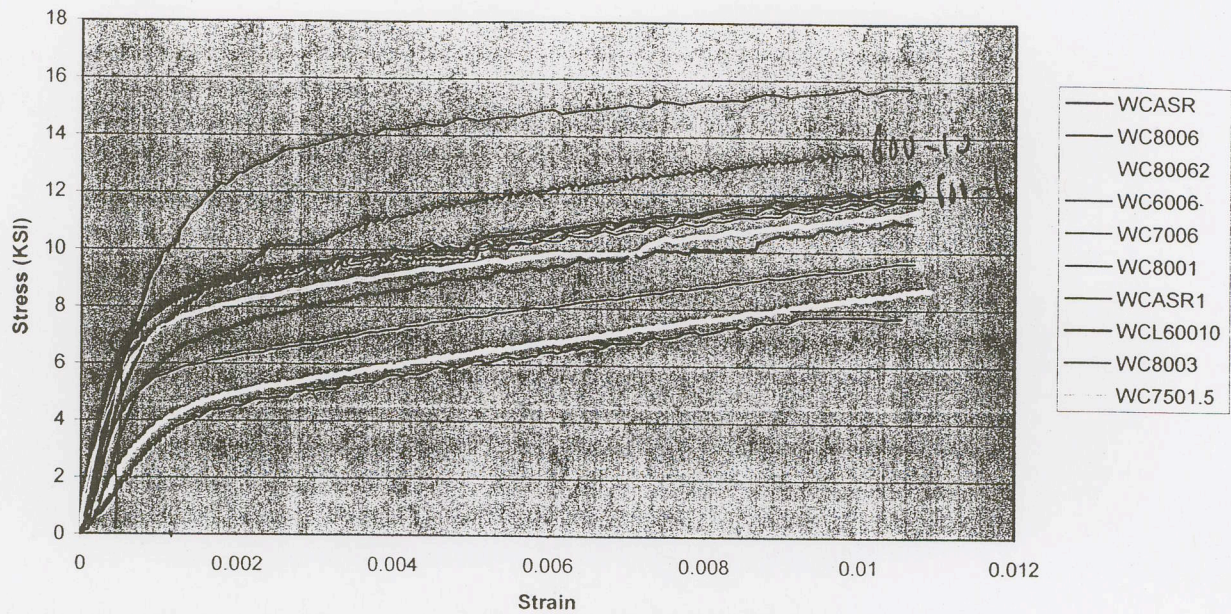


Figure 2(a) Stress - Strain curves with different heat treatments

SNS high RRR niobium

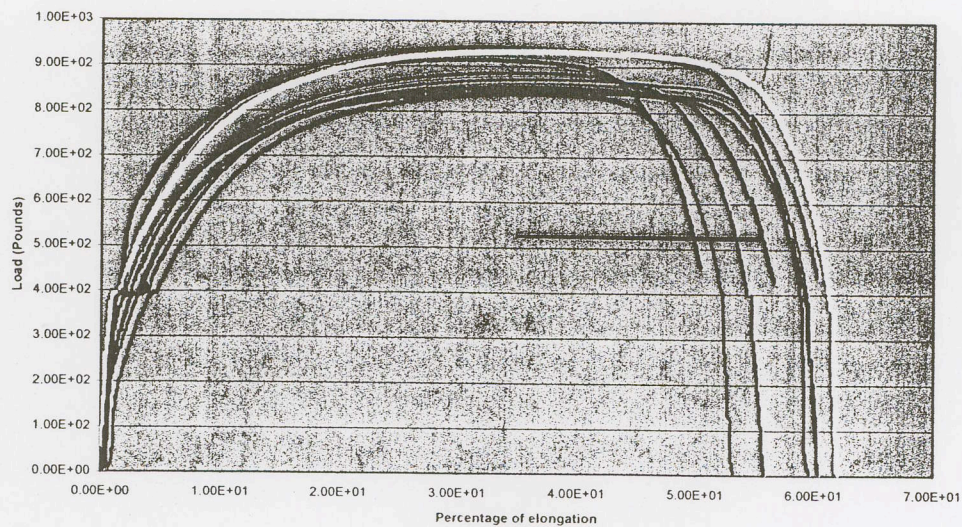


Figure 2(b) Load - Percentage of elongation for different heat treatments

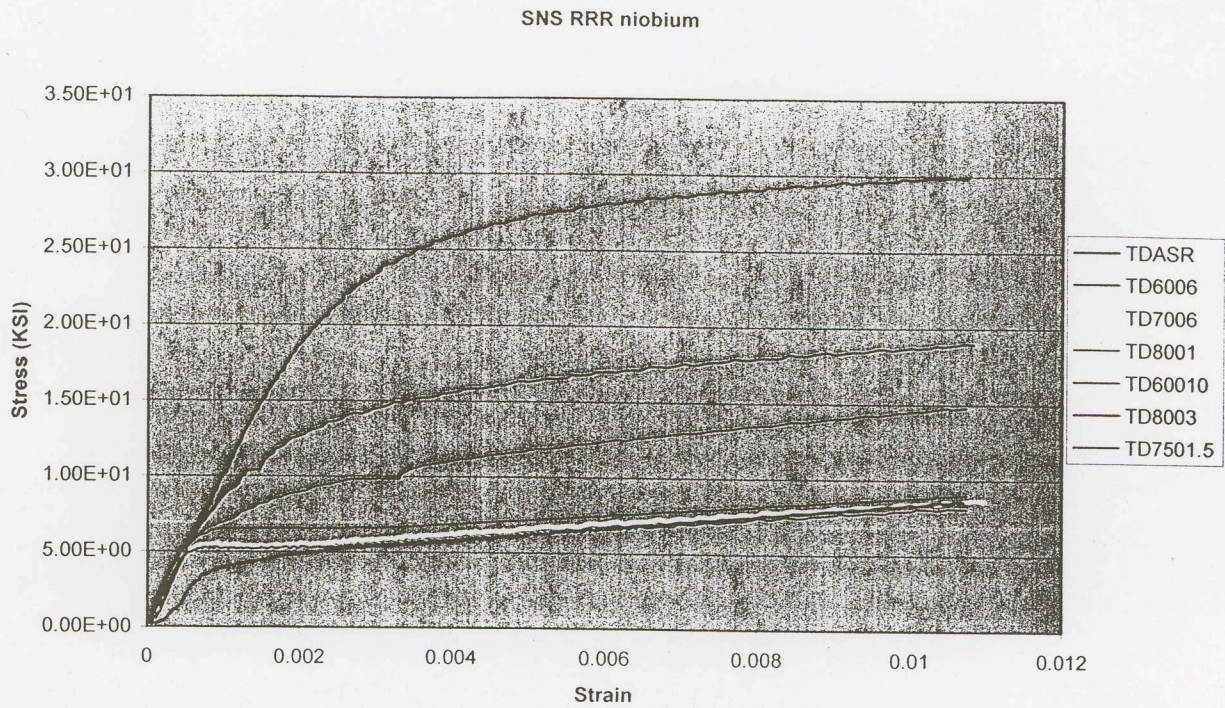


Figure 3(a) Stress - Strain curves with different heat treatments

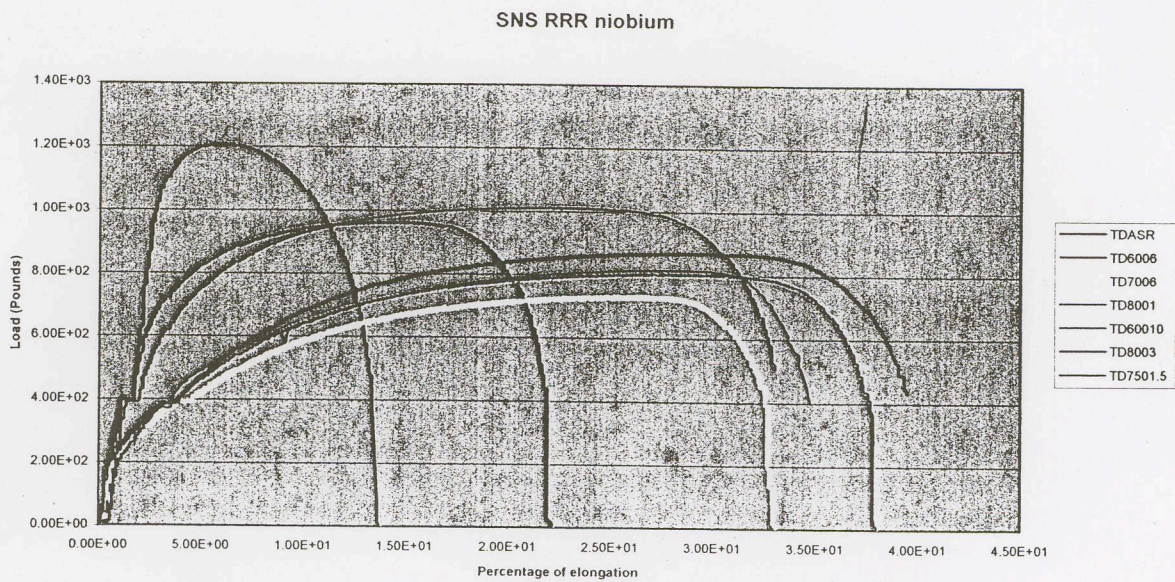


Figure 3(b) Load - Percentage of elongation for different heat treatments

Table I

Summary of the mechanical & thermal properties of SNS niobium

Sample No.	Yield Strength (KSI)	Tensile Strength (KSI)	% Elongation	RRR	Remarks
1	12.32	21.98	61	450	WCLASR1
2	10.9	19.42	56	490	WCLASR2
3	11	20.6	56	310	WCLASR3
4	11.2	21.39	54	341	WCLASR4
5	11.3	21.66	56	460	WCLASR5
6	10.5	24.4	60		WCLASR6
7	9.4	25.7	65		WCLASR7*
8	5.5	21.1	53		WCL80061
9	6.0	21.5	60		WCL80062
10	5.9	23.6	50		WCL8003*
11	6.0	22.4	60		WCL8003
12	7.0	22.8	50		WCL8001
13	7.7	23.7	61		WCL7501.5
14	7.5	24.2	55		WCL7006
15	9.5	26.3	57		WCL60010
16	10.5	22.6	54		WCL6006
17	14	23.4	51		WCASR1
18	13	23.8	41		WCASR2*
19	6.5	21.4	59		WC8003
20	7.1	21.5	--		WC8003*

21	8.1	22.4	60	WC8001
22	9.2	21.8	61	WC7006
23	8.8	22.3	60	WC7501.5
24	10.5	22.7	57	WC60010
25	9.0	22.9	62	WC6006
26	27	31	14	TDASR1
27	22.6	31.2	6.6 ⁺	TDASR2*
28	5.5	22.8	41	TD8003*
29	5.5	22	40	TD8003
30	5.5	20.6	38	TD8001
31	6.6	21.9	35	TD7501.5
32	5.6	19.2	33	TD7006
33	10	25	34	TD60010
34	15	25	22	TD6006

Note: WCL - SNS prototype; WC - SNS production high RRR; TD - SNS production RRR

* Materials Research & Engineering, Inc carried out these measurements.

+ Sample failed near gage mark, percentage of elongation may be higher

-- Sample failed at the radius, near the tab. Elongation data is not valid

Figures 4, 5 and 6 show the optical micrographs of the WCL, WC and TD RRR niobium⁷. From these micrographs it can be inferred that WCL and WC are completely recrystallized whereas TD niobium is not at all recrystallized. Further WCL is homogenous with uniform grains while WC is not completely homogeneous and shows different size grains. It is well known that the recrystallization process is influenced by purity of the material, the nature (cross-rolled or not) and amount of cold work (percentage of deformation), and the annealing temperature and duration of annealing⁸. Since we do not have the information on all these

parameters of the tested materials, it would be rather difficult to obtain a clear picture of what is happening with various heat treatments the materials were subjected to.

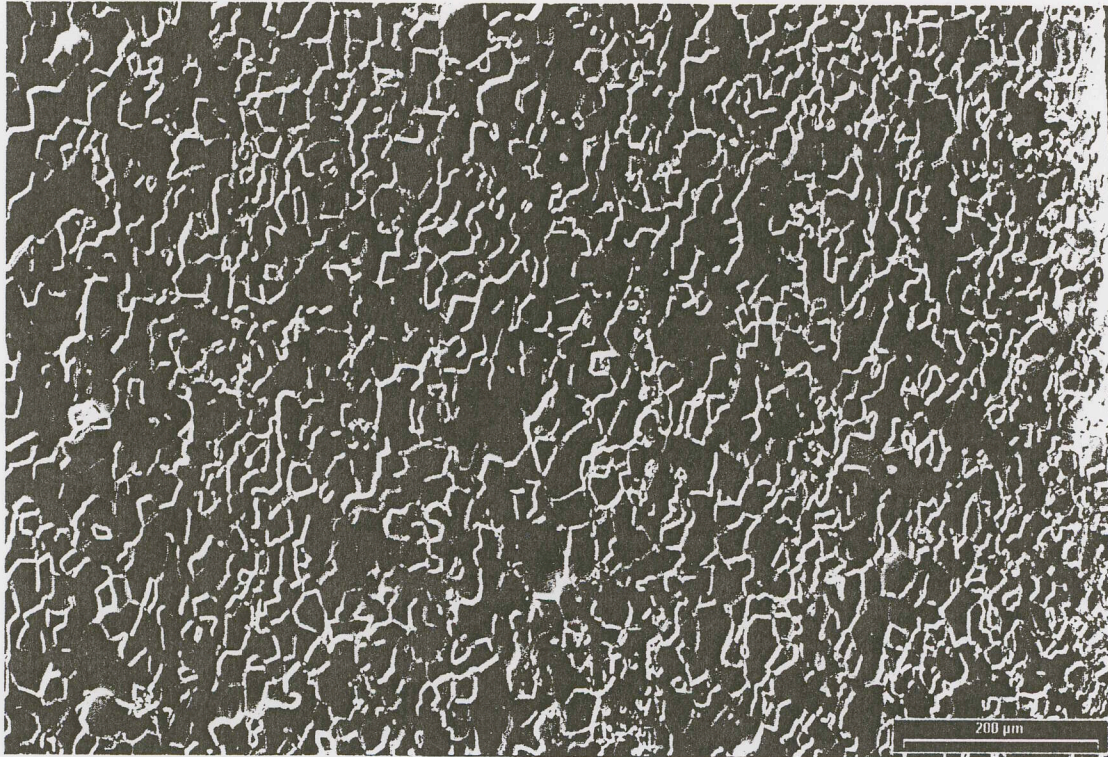


Figure 4. Micrograph of WCL niobium



Figure 5. Micrograph of WC niobium

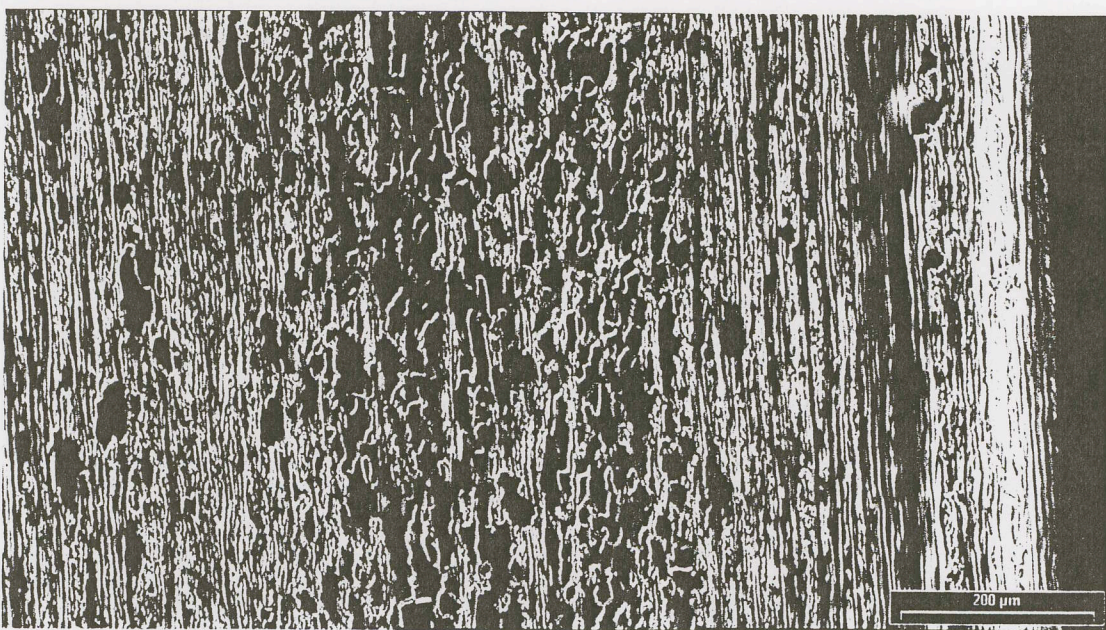


Figure 6. Micrograph of TD niobium

The yield strength starts decreasing for all batches of niobium as expected with increasing heat treatment temperature and appears to change considerably at temperatures higher than 600 C and is summarized in figure 7. The ductility (extension before break) for all the three batches of niobium is shown in Figure 8 (note: 650 on the x-axis corresponds to sample 60010; similarly, 850 corresponds to 8003 and 900 corresponds to 8006). The ductility of both WCL and WC niobium improved with increasing heat treatment temperature as expected, and as received niobium data agrees with vendor's data. The ductility of the as received TD niobium is very low, and it is understandable because this material is not recrystallized at all after the cold work. Vickers hardness of these materials is shown in figure 9. The apparent reduction in Vickers hardness initially with heat treatment and subsequent increase at higher temperature seems to have also been observed by other investigators⁹. Figure 10 summarizes the yield strength relationship with Vickers hardness. A detailed discussion on the apparent reduction of Young's modulus at high heat treatment temperatures in the case of WC and TD will be presented later on completing the ongoing study with various batches of niobium from a single manufacturer.

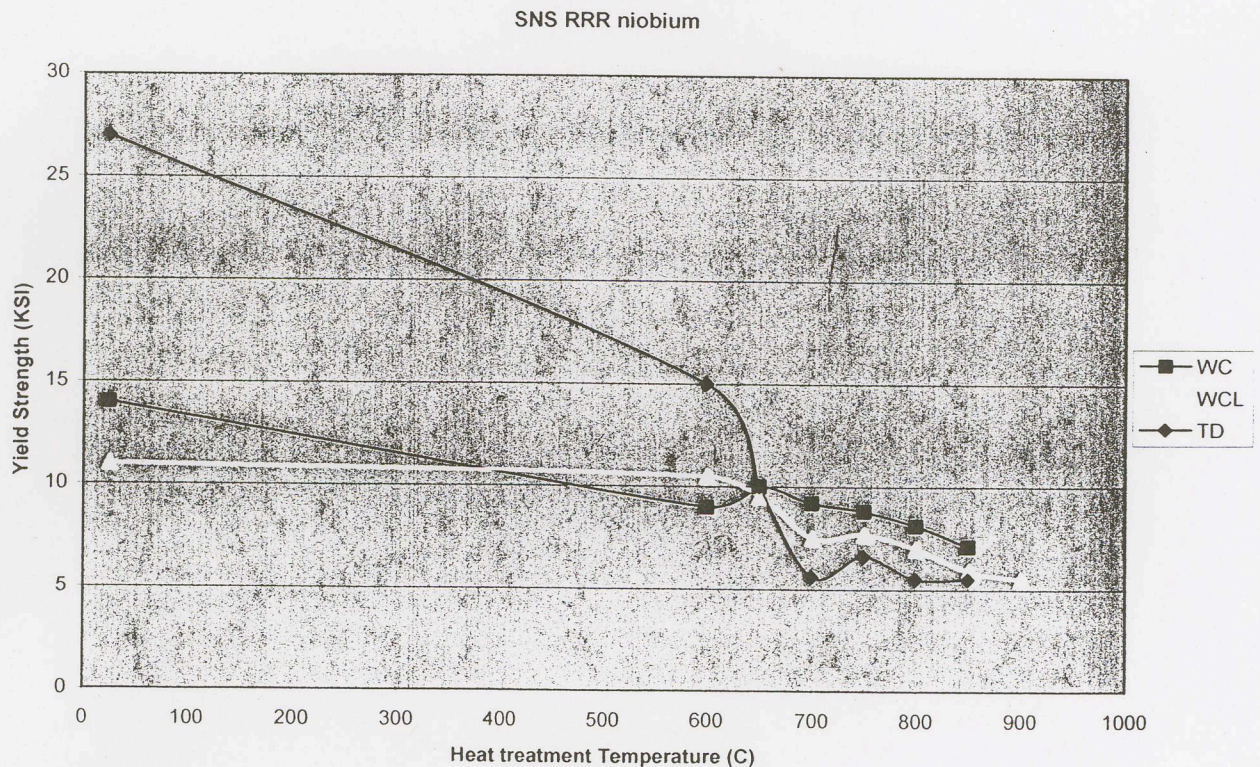


Figure 7. Yield strength as a function of heat treatment temperature

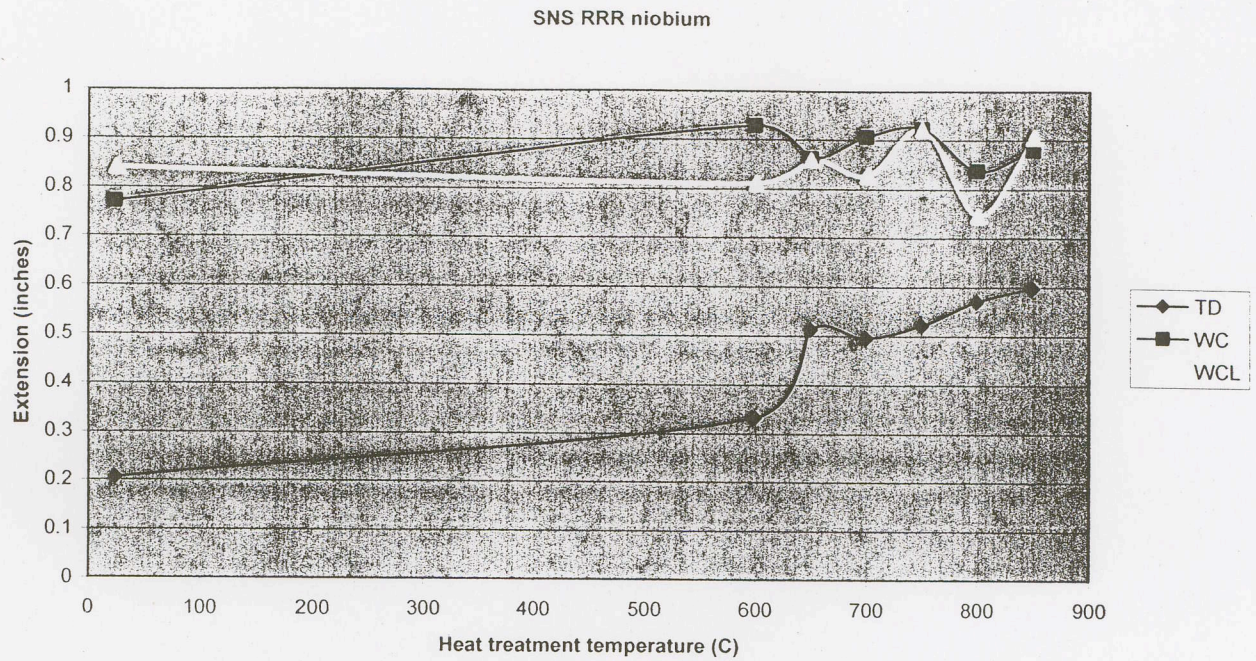


Figure 8. Extension at break as a function of heat treatment temperature

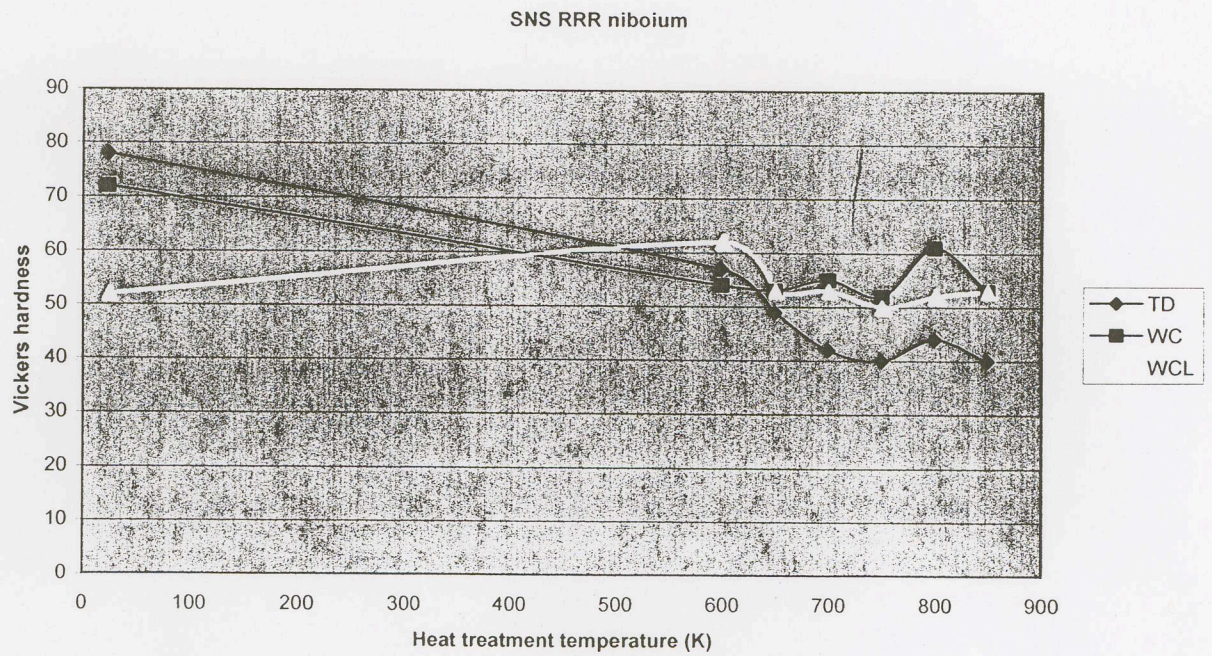


Figure 9. Vickers hardness as a function of heat treatment temperature

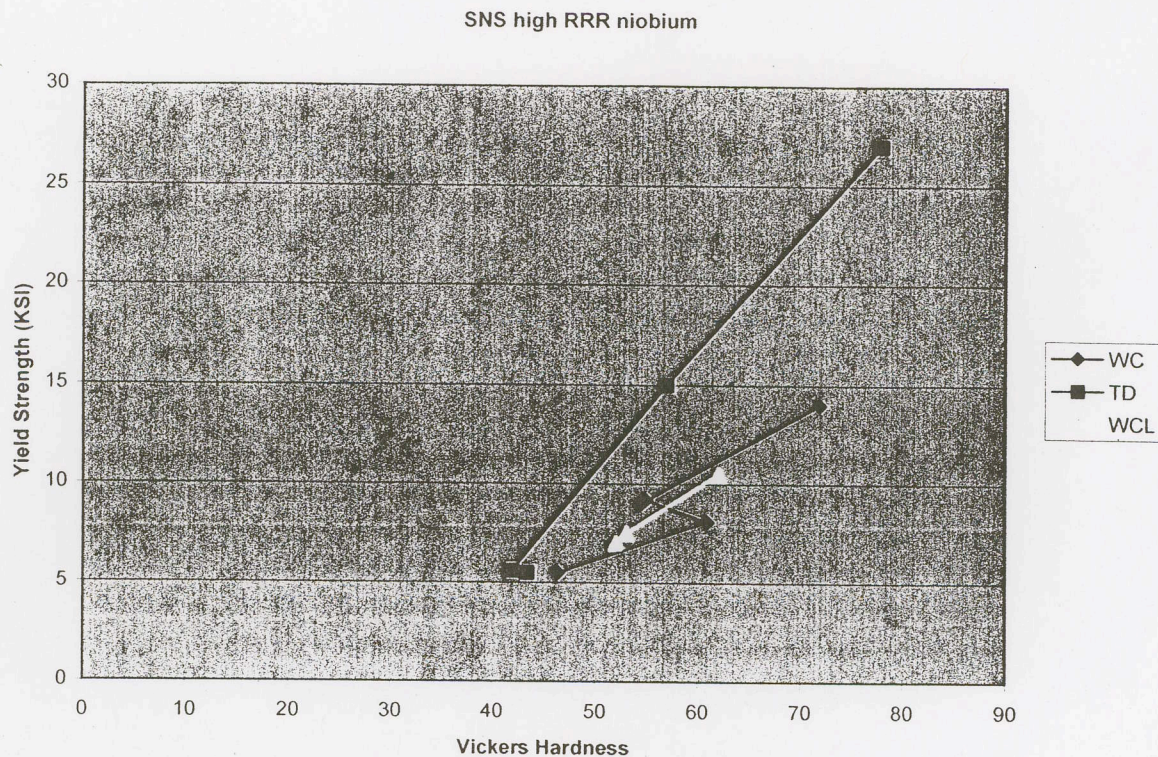


Figure 10. Yield strength as a function of Vickers hardness

References:

1. G. Ciovati, Private Communication
2. M. G. Rao and P. Kneisel, Mechanical Properties of high RRR Niobium, in R&D Issues in the Field of Superconducting Cavities, Edited by D. Proch, DESY Print May 1995, TESLA 95-09 pp. 85-122
3. P. Kneisel et al., "Proc. of the Conf. Electron Beam Melting and Refining State of the Art 1990," R. Bakish Ed.; Bakish Materials Corporation, Englewood, 1990 pp. 177-188
4. M. G. Rao and P. Kneisel, Advances in Cryogenic Engineering, Vol. 40 Edited by R. P. Reed et al., Plenum Press, New York, 1994 pp. 1383-1390
5. Hasan Padamsee et al., RF Superconductivity for Accelerators, John Wiley & Sons Inc., New York, 1998 pp. 109-113
6. M. G. Rao et al., in Proceedings of the 6th Workshop on RF Superconductivity, Edited by R. M. Sundelin, CEBAF, Newport News, Va 1994 pp. 643-649
7. Xenia Singer, Private Communication
8. D. A. Wigley, Mechanical Properties of Materials at Low Temperatures, Plenum Press, New York 1971 pp. 41
9. Craig Wojcik, Private Communication
10. W. Singer, Private Communication